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AGRICULTURE

CIRCULAR No. 4.—(To accompany Bulletin No. 46.)

United States Department of Agriculture,

DIVISION OF CHEMISTRY.

WASHINGTON, D. C., *December 11, 1897.*

All the changes in methods of analysis of the Association of Official Agricultural Chemists, which were made at the annual meetings of 1896 and 1897, have been incorporated in Circular No. 4. In this circular, the pages in the edition of Bulletin 46, where the methods described would naturally be found, are indicated. By this arrangement, the expense of entirely reprinting Bulletin 46 has been avoided without sacrificing, to any appreciable extent, the availability of the methods.

This circular will be furnished to all members of the Association of Official Agricultural Chemists and to others in so far as the extent of the edition will permit.

H. W. WILEY,
Chief of Division, Secretary A. O. A. C.

Approved:
JAMES WILSON,
Secretary.

CHANGES IN AND ADDITIONS TO METHODS OF ANALYSIS ADOPTED AT THE THIRTEENTH AND FOURTEENTH ANNUAL MEETINGS OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

[In all cases, unless otherwise mentioned, pages and paragraphs cited refer to Bulletin No. 46, Chemical Division of the United States Department of Agriculture.—H. W. W.]

DETERMINATION OF PHOSPHORIC ACID.

Page 14 (2) (*b*) (b_1), eighth line, omit "six," and insert "from ten to fifteen."

Sixteenth line, omit all after "wash" to end of sentence and insert "from five to six times with water, using in all from 150 to 250 cc."

5. DETERMINATION OF POTASH.

(Insert instead of methods on pages 22, 23, and 24.)

(a) LINDO-GLADDING METHOD.

(1) *Preparation of reagents.*

(*a*) *Ammonium chlorid solution.*—Dissolve 100 grams of ammonium chlorid in 500 cc of water, add from 5 to 10 grams of pulverized potassium platinic chlorid, and shake at intervals for six or eight hours. The mixture is then allowed to settle over night and filtered, and the residue is ready for the preparation of a fresh supply when needed.

(*b*) *Platinum solution.*—The platinum solution used contains 1 gram of metallic platinum (2.1 grams of H_2PtCl_6) in every 10 cc.

(2) *Methods of making solution.*

(*a*) *With potash salts and mixed fertilizers.*—Boil 10 grams of the sample with 300 cc of water thirty minutes. In the case of mixed fertilizers, add to the hot solution a slight excess of ammonia and then sufficient powdered ammonium oxalate to precipitate all the lime present. Cool, dilute to 500 cc, mix and pass through a dry filter. In case of muriate and sulfate of potash, sulfate of potash and magnesia and kainit, dissolve and dilute to 500 cc without the addition of ammonium and ammonium oxalate.

(*b*) *With organic compounds.*—When it is desired to determine the total amount of potash in organic substances like cotton seed meal, tobacco stems, etc., saturate 10 grams with strong sulfuric acid, and ignite in a muffle at a low red heat to destroy organic matter. Add a little strong hydrochloric acid and warm slightly in order to loosen the mass from the dish, and proceed as directed under (3) (*a*).

(3) *Determination.*

(*a*) *In mixed fertilizers.*—Evaporate 50 cc of the solution made according to (2), corresponding to 1 gram of the sample, nearly to dryness, add 1 cc of dilute sulfuric acid (1 to 1), evaporate to dryness and ignite to whiteness. As all the potash is in form of sulfate, no loss need be apprehended by volatilization of potash, and a full red heat must be maintained until the residue is perfectly white. Dissolve the residue in hot water, using at least 20 cc for each decigram of K_2O, add a few drops of hydrochloric acid and platinum solution in excess. Evaporate on a water bath to a thick paste and treat the residue with 80 per cent alcohol, sp. gr. 0.8645, avoiding the absorption of ammonia. Wash the precipitate thoroughly with 80 per cent alcohol both by decantation and on the filter, continuing the washing after the filtrate is colorless. Wash finally with 10 cc of the ammonium chlorid solution (1) (*a*), to remove impurities from the precipitate and repeat this washing five or six times. Wash again thoroughly with 80 per cent alcohol and dry the precipitate for thirty minutes at 100°. The precipitate should be perfectly soluble in water.

(*b*) *Muriate of potash.*—Dilute 25 cc of the solution, prepared according to (2) (a), with 25 cc of water, acidify with a few drops of hydrochloric acid, add 10 cc of platinum solution and evaporate to a thick paste. Treat the residue as under (3) (a).

(*c*) *Sulfate of potash, sulfate of potash and magnesia, and kainit.*—Dilute 25 cc of the solution, prepared according to (2) (a), with 25 cc of water, acidify with a few drops of hydrochloric acid and add 15 cc of platinum solution. Evaporate the mixture and proceed as directed under (3) (a), except that 25 cc portions of ammonium chlorid solution should be used.

(b) Optional Method.*

(1) *Preparation of reagent.*

Platinum solution.—The platinum solution used is the same as that described under the Lindo-Gladding method.

(2) *Method of making solution.*

The solution is prepared as directed under the Lindo-Gladding method, omitting in all cases the addition of ammonia and ammonium oxalate.

* The Lindo-Gladding method is preferable in the presence of soluble sulfates.

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(3) *Determination.*

Dilute 25 cc of the solution made as directed under (2), (50 cc, if less than 10 per cent of potassium oxid be present) to 150 cc, heat to 100°, and add, drop by drop with constant stirring, a slight excess of barium chlorid solution. Without filtering, in the same manner add barium hydrate in slight excess. Filter while hot and wash until the precipitate is free from chlorids. Add to the filtrate 1 cc of strong ammonium hydrate, and then a saturated solution of ammonium carbonate until the excess of barium is precipitated. Heat and add, in fine powder, 0.5 gram of pure oxalic acid or 0.75 gram of ammonium oxalate. Filter and wash free from chlorids, evaporate the filtrate to dryness in a platinum dish, and ignite carefully over the free flame below a red heat until all volatile matter is driven off. Digest the residue with hot water, filter through a small filter and dilute the filtrate, if necessary, so that for each decigram of K_2O there will be at least 20 cc of liquid. Acidify with a few drops of hydrochloric acid and add platinum solution in excess. Evaporate on a water bath to a thick sirup and treat the residue with 80 per cent alcohol (specific gravity 0.8645). Wash the precipitate thoroughly with 80 per cent alcohol both by decantation and after collecting on a Gooch crucible or other form of filter. Dry for thirty minutes at 100° and weigh.

It is desirable, if there be an appearance of foreign matter in the double salt, that it should be washed according to the previous method with several portions of the ammonium chlorid solution of 10 cc each.

(c) Factors.

For the conversion of potassium platinichlorid to KCl use the factor 0.3069, to K_2SO_4 use 0.3587, and to K_2O, 0.1939.

METHODS FOR THE ANALYSIS OF DAIRY PRODUCTS.

Provisional Method of Saponification.

Insert on page 28, after (b_1), the following:

Using the same amounts of alkali and alcohol as described above, the saponification may be conducted on a steam or water bath, the flask being connected with a reflux condenser consisting of a glass tube of not less than one meter in length, or of a shorter condenser cooled by a current of water.

Page 36, under 2, *Milk Analysis;* omit first and second sentences and insert:

1. Heat to constant weight at the temperature of boiling water from 1 to 2 grams of milk in a tared flat dish of not less than 5 cc diameter. If desired, from 15 to 20 grams of pure dry sand may be previously placed in the dish.

Insert 2 before "*Babcock Asbestus Method.*"

Page 36 (c). Determination of Total Nitrogen.

Add at end of paragraph the following:

1. Provisional method for determination of casein in cow's milk.—The determination of casein in milk should be made when the milk is fresh, or nearly so. When it is not practicable to make this determination within twenty-four hours, add 1 part of formaldehyd to 2,500 parts of milk, and keep in a cool place. Weigh about 10 grams of milk, dilute in a beaker with about 90 cc of water at 40°–42°, and add at once 1.5 cc of a solution containing 10 per cent of acetic acid by weight. Stir with a glass rod, and let stand from three to five minutes longer. Then decant on filter, wash two or three times with cold water by decantation, and transfer precipitate completely to filter. Wash once or twice on filter. The filtrate should be clear, or very nearly so. If it be not clear when

it first runs through, it can generally be made so by two or three repeated filtrations, after which the washing of the precipitate can be completed. The washed precipitate and filter paper are digested as in the regular Kjeldahl method for the determination of nitrogen, and the process is completed as usual. To calculate the nitrogen into an equivalent amount of casein, multiply by 6.25.

In working with milk which has been kept with preservatives, the acetic acid should be added in small proportions, a few drops at a time, with stirring and the addition continued until the liquid above the precipitate becomes clear, or very nearly so.

2. Provisional method for determining albumin in milk.—The filtrate obtained in the above operation is neutralized with caustic alkali, three-tenths cc of a 10 per cent solution of acetic acid added and the mixture heated to the temperature of boiling water for from ten to fifteen minutes. The precipitate is collected on a filter, washed, and the nitrogen therein determined. Nitrogen multiplied by 6.25 equals albumin.

3. Provisional optional method for determining casein in milk.—To 5 grams of milk, 50 cc of a solution of magnesium sulfate saturated at from 40° to 45° are added and the mixture heated to about 45°, until the precipitate separates and subsides, leaving the supernatant liquid clear. The precipitate is collected on a filter, washed two or three times with the solution of magnesium sulfate prepared as above and at the same temperature, viz, about 45°, and the nitrogen therein determined.

4. Provisional optional method for determining albumin in milk.—To the filtrate, obtained by the above process, are added three-tenths cc of a 10 per cent solution of acetic acid and the mixture boiled until the albumin is completely precipitated. The precipitate is collected on a filter, washed as above described, and the nitrogen therein determined.

Page 37, "3. Cheese Analysis (Provisional Methods)," omit "(Provisional Methods)."

Page 37, omit all of (a) save caption and insert the following:

When the cheese can be cut, a narrow wedge-shaped segment reaching from the outer edge to the center of the cheese is taken. This is to be cut into strips and passed through a sausage-grinding machine three times. When the cheese can not be cut, samples are taken by a cheese trier. If only one plug can be obtained, this should be taken perpendicular to the surface at a point one-third of the distance from the edge to the center of the cheese. The plug should reach either entirely through or only halfway through the cheese. When possible, draw three plugs—one from the center, one from a point near the outer edge, and one from a point halfway between the other two. For inspection purposes, the rind may be rejected; but for investigations requiring the absolute amount of fat in the cheese the rind is included in the sample. It is preferable to grind the plugs in a sausage machine, but when this is not done they are cut very fine and carefully mixed.

Page 37, under (b), omit first and second lines and "tus" of the third line and insert:

From 2 to 5 grams of cheese should be placed in a weighed platinum or porcelain dish which contains a small quantity of material like freshly ignited asbestus, sand, etc.

Page 37, omit under (c) the whole paragraph and insert:

The extraction tube is prepared as follows:

Cover the perforations in the bottom of the tube with asbestus, and on this place a mixture containing equal parts of anhydrous copper sulfate and pure, dry sand to the depth of about 5 cm, packing loosely. Cover the upper surface

of this material with a film of asbestus. On this are placed from 2 to 5 grams of the sample of cheese. The tube is placed in a continuous extraction apparatus and treated for five hours with anhydrous ether. The cheese is removed and ground to a fine powder with pure sand in a mortar. The mixed cheese and sand are replaced in the extraction tube, the mortar washed free of all matters with ether, the washings being added to the tube, and the extraction is continued for ten hours.

Page 37, add: (g) *Provisional method for the determination of acidity in cheese.*—Add water, at a temperature of 40°, to 10 grams of finely divided cheese until the volume equals 105 cc; agitate vigorously and filter. Titrate portions of 25 cc of filtrate, corresponding to 2.5 grams of cheese, with a standardized solution of sodium hydroxid, preferably one-tenth normal. Use phenolphtalein as indicator. Express amount of acid as lactic acid.

OPTIONAL METHOD OF DETERMINING PHOSPHORIC ACID IN SOILS.

NOTE.—The following method, devised by Mr. Goss, was recommended as an optional method by the association in 1895, but was not ordered printed with the official methods. A synopsis of it is printed here for the convenience of analysts:

Page 41, after (g), insert the following:

"Ten grams of the air-dried soil, passed through a sieve of one millimeter mesh, are placed in a small Kjeldahl flask marked at 250 cc. From 20 to 30 cc concentrated sulfuric acid and approximately 0.7 gram yellow oxid of mercury are added, the contents of the flask well mixed by shaking, and oxidized over the open flame, as in the determination of nitrogen, for an hour. After cooling, about 100 cc of water, 5 cc of concentrated hydrochloric acid and 2 cc of concentrated nitric acid are added, and the mixture reboiled to oxidize the iron, cooled, the volume completed to one-fourth of a liter with water, and the contents of the flask filtered through a dry, folded filter paper. One hundred cubic centimeters of the filtrate are placed in a flask of about 450 cc capacity, strong ammonia added until a permanent precipitate is formed, which is dissolved by the addition of about 7 cc of nitric acid, and the mixture boiled until clear. The flask is removed from the flame and cooled at room temperature for exactly two minutes, 75 cc molybdate solution added, and the flask placed in a water bath kept at 80° for 15 minutes, shaking vigorously four or five times meanwhile. After removing from the bath, the flask is allowed to stand for ten minutes until the precipitate has settled, and the supernatant liquid is poured on to the filter paper under pressure, the precipitate being partially brought upon the paper. The flask and precipitate are thoroughly washed with ammonium-nitrate solution, the precipitate either by decantation or in the filter paper. The flask is then placed under the filter, the precipitate is dissolved in ammonia, and the phosphoric acid estimated by the usual processes. Details of the manipulation are given in Bulletin No. 43 of the Division of Chemistry, pp. 58–60."

Page 42, omit all of 8 and insert:

From 7 to 14 grams of the soil are placed in a small Kjeldahl digesting flask, about 250 cc capacity, with 30 cc of strong sulfuric acid, or more, if necessary, and 0.7 gram yellow oxid of mercury, and boiled for an hour. The residue is oxidized with potassium permanganate in the usual way. After cooling, the flask is half filled with water, vigorously shaken, the heavy matters allowed to subside and the supernatant liquid poured into a flask of from 1,000 to 1,200 cc capacity. This operation is repeated until the ammonium sulfate is practically

all removed and the large flask is a little more than half full and the ammonia distilled in the usual manner. If a sample be known to contain a considerable amount of nitrate, use method p. 17 (*c*).

Page 54, first line, read pages "55–57," instead of page 55.

METHODS FOR THE ANALYSIS OF FOODS AND FEEDING STUFFS.

Page 62. Read caption in (c) DETERMINATION OF STARCH IN COMMERCIAL STARCHES.

Omit 3 in first line of (c) and insert 2.5.

Add after section (c), page 62, the following:

(c_1) DIASTASE METHOD FOR STARCH.

Extract from 2 to 5 grams of the finely powdered substance with ether, bring the extracted residue on to a filter, or into a Gooch crucible, and wash with 150 cc of 10 per cent alcohol, and then with a little strong alcohol. Place the residue in a beaker with 50 cc of water, immerse the beaker in a boiling water bath, and stir constantly until all the starch is gelatinized, cool to 55°, add from 20 to 40 cc of malt extract and maintain at this temperature until the solution no longer gives the starch reaction with iodin: Cool and make up directly to 250 cc, filter, bring 200 cc of the filtrate into a ten ounce flask with 20 cc of 25 per cent hydrochloric acid (sp. gr. 1.125), connect with a reflux condenser and boil for two hours and a half, exactly neutralize while hot with sodium carbonate, avoiding excess, cool and make up to 500 cc. Mix the solution well, pour through a dry filter, and determine the dextrose in an aliquot part by Allihn's method, pages 52–57. Convert the starch into dextrose by the factor .90.

Add on page 62, after (c_1), the following:

(c_2) METHOD FOR ESTIMATING GALACTAN.

Bring three grams of the substance into a beaker about 5.5 cm in diameter and 7 cm deep, together with 60 cc of nitric acid of 1.15 specific gravity and evaporate the solution to exactly one-third of its volume on a water bath at a temperature of 94 to 96°. After standing twenty-four hours, add 10 cc of water to the precipitate, and allow it to stand another twenty-four hours. The mucic acid has in the meantime crystallized, but is mixed with considerable material only partially oxidized by the nitric acid. The solution is therefore filtered through filter paper, washed with 30 cc of water, to remove as much of the nitric acid as possible, and the filter and contents brought back into the beaker. Thirty cc of ammonium carbonate solution consisting of one part ammonium carbonate, 19 parts water, and 1 part strong ammonia, are added, and the beaker brought onto a water bath and heated gently for fifteen minutes. The ammonium carbonate takes up the mucic acid, forming the soluble muciate of ammonia. The solution is filtered into a platinum or porcelain dish, and the residue thoroughly washed with water to remove all of the muciate of ammonia. The filtrate is evaporated to dryness over a water bath, 5 cc of nitric acid of 1.15 specific gravity are added, and the mixture thoroughly stirred and allowed to stand for thirty minutes. The nitric acid decomposes the ammonium muciate, precipitating the mucic acid, which is collected on a tared filter or Gooch crucible, washed with from 10 to 15 cc of water, then with 60 cc of alcohol and quite a number of times with ether, dried at 100° for a short time, and weighed. The mucic acid multiplied by 1.33 gives galactose, and the product multiplied by .9 gives galactan.

Page 63, omit at end of (d) all relating to factors and insert:

The weight of hydrazone $\times$ 0.516 + 0.0104 $\div$ weight of sample used = percentage furfurol.

Furfurol $\times$ 1.84 = pentosans.

Pentosans $\div$ 0.88 = pentoses.

Furfurol $\times$ 1.65 = xylan.

Furfurol $\times$ 2.03 = araban.

Determination of Pentosans by means of Phloroglucin.

On page 63, at the conclusion of (d) add the following:—(d_1) Three grams of the material are brought into a ten-ounce flask, together with 100 cc of 12 per cent hydrochloric acid (sp. gr. 1.06) and several pieces of recently heated pumice stone. The flask, placed upon wire-gauze, is connected with a Liebig condenser, and heat applied, rather gently at first, using a gauze top to distribute the flame, and so regulated as to distill over 30 cc in about ten minutes. The 30 cc driven over are replaced by a like quantity of the dilute acid by means of a separatory funnel, and the process continued as long as the distillate gives a pronounced reaction with aniline acetate on filter paper. To the completed distillate is gradually added a quantity of pholoroglucin dissolved in 12 per cent hydrochloric acid, and the resulting mixture thoroughly stirred. The amount of phloroglucin used should be about double that of the furfurol expected. The solution first turns yellow, then green; and very soon an amorphous greenish precipitate appears, which grows rapidly darker, till it finally becomes almost black. The solution is made up to 500 cc with 12 per cent hydrochloric acid, and allowed to stand over night. In case there is very little furfurol in the substance tested, and the resulting distillate consequently small, it is best to add sufficient 12 per cent hydrochloric acid to the distillate before adding the phloroglucin solution, so that, upon the addition of the latter solution, the resulting mixture will contain approximately 500 cc.

The amorphous black precipitate is filtered into a tared Gooch crucible through an asbestus felt, washed with 100 cc of water, dried to constant weight by heating from three to four hours at 100°, cooled and weighed, the increase in weight being reckoned as phloroglucid. To calculate the furfurol from the phloroglucid, use the following table:

Weight of phloroglucid:

.20 gram		1.820	
.22 "		1.839	
.24 "		1.856	
.26 "		1.871	
.28 "		1.884	
.30 "		1.895	
.32 "	$\div$	1.904	= furfurol.
.34 "		1.911	
.36 "		1.916	
.38 "		1.919	
.40 "		1.920	
.45 "		1.927	
.50+ "		1.930	

METHODS FOR THE ANALYSIS OF FERMENTED AND DISTILLED LIQUORS.

Add on page 77, after 18:

"19. Determination of Fusel Oil.

"The apparatus recommended for this determination is Bromwell's modification of Roese's fusel-oil apparatus.

"This apparatus consists of a pear-shaped bulb holding about 200 cc, stoppered at the upper end and sealed at the lower to a graduated stem about 4 mm in internal diameter. To the lower end of this graduated stem is sealed a bulb of 20 cc capacity, the lower end of which bears a stopcock tube. The apparatus is graduated to 0.02 cc, from 20 cc to 22.5 cc.

"The reagents required are fusel-free alcohol that has been prepared by fractional distillation over caustic soda or caustic potash, and diluted to exactly 30 per cent by volume (sp. gr., 0.96541), chloroform freed from water and redistilled, and sulfuric acid (sp. gr., 1.2857 at 15.6°).

"Distill slowly 200 cc of the sample under examination till about 175 cc have passed over, allow the distilling flask to cool, add 25 cc of water, and distill again till the total distillate measures 200 cc. Dilute the distillate to exactly 30 per cent by volume (sp. gr., 0.96541 at 15.6°).

"The following is an accurate method for diluting any given alcohol solution to a weaker solution of definite percentage: Designate the volume percentage of the stronger alcohol by V, and that of the weaker alcohol by v. Mix v volumes of the stronger alcohol with water to make V volumes of the product. Allow the mixture to stand till full contraction has taken place, and till it has reached the temperature of the original alcohol and water, and make up any deficiency in the V volumes with water.

"*Example.*—It is desired to dilute a distillate containing 50 per cent of alcohol by volume until it contains 30 per cent. To 30 volumes of the 50 per cent alcohol add enough water to make 50 volumes, or place 150 cc of the distillate in a 250 cc flask, fill to the mark with water, mix, cool, and fill to the mark again.

"Prepare a water bath, the contents of which are kept at exactly 15°, and place in it the apparatus (covering the end of the tube with a rubber cap to prevent wetting the inside of the tube), and flasks containing the 30 per cent fusel-free alcohol, chloroform, sulfuric acid, and the distillate diluted to 30 per cent by volume. When the solutions have all attained the temperature of 15°, fill the apparatus to the 20 cc mark with the chloroform, drawing it through the lower tube by means of suction, add 100 cc of the 30 per cent fusel-free alcohol and 1 cc of the sulfuric acid, invert the apparatus, and shake vigorously for two or three minutes, interrupting once or twice to open the stopcock for the purpose of equalizing pressure. Allow the apparatus to stand ten or fifteen minutes in water that is kept at the temperature of 15°, turning occasionally to hasten the separation of the reagents, and note the volume of the chloroform. After thoroughly cleansing and drying the apparatus, repeat this operation, using the diluted distillate from the sample under examination in place of the fusel-free alcohol. The increase in the chloroform volume with the sample under examination over that with the fusel-free alcohol is due to fusel oil, and this difference (expressed in cubic centimeters), multiplied by the factor 0.663, gives the volume of fusel oil in 100 cc, which is equal to the percentage of fusel oil by volume in the 30 per cent distillate. This must be calculated to the percentage of fusel oil by volume in the original liquor.

"*Example.*—A sample of liquor contains 50 per cent of alcohol by volume. The increase in the chloroform volume with the 30 per cent fusel-free alcohol is 1.42 cc; the increase in the chloroform volume with the distillate from the liquor under examination, diluted to 30 per cent, is 1.62 cc: difference, 0.20 cc. The volume of fusel oil in 100 cc of the 30 per cent distillate then is $0.20 \times 0.663 = 0.1326$ cc, and by the proportion $30 : 50 :: 0.1326 : 0.221$, we obtain the percentage of fusel oil by volume in the original liquor.

"20. Determination of Aldehyds.

"(a) *Preparation of reagent.*—Eighty cubic centimeters of a saturated solution of sodium disulfite are mixed with a solution of 0.12 gram of fuchsin in about 800 cc of water, 12 cc of sulfuric acid added, the solution thoroughly mixed, and diluted with water to 1 liter.

"(b) *Determination.*—A portion of the sample is diluted with water, or strengthened with aldehyd-free alcohol until it contains 50 per cent of alcohol by volume, and 25 cc of this solution are treated with 10 cc of the reagent, and allowed to stand twenty minutes. At the same time 25 cc of a solution of 0.05 gram of acetic aldehyd in 1,000 cc of 50 per cent alcohol are treated in the same manner and allowed to stand the same length of time. The relative intensity of the colors of the two solutions is then determined by means of a colorimeter, and from the figure thus obtained the weight of aldehyd is estimated as acetic aldehyd, and calculated to percentage of the original liquor.

"21. Determination of Ethereal Salts.

"After the determination of the volatile acids, the neutralized distillate is transferred to a flask connected with a reflux condenser, treated with 25 cc of tenth normal sodium hydroxid, and boiled one-half hour. The flask and contents are then cooled, 25 cc of tenth normal hydrochloric acid added, and the excess of acid titrated with sodium hydroxid, using phenolphtalein as indicator. The number of cubic centimeters of tenth normal alkali used in this titration, multiplied by 0.0088, is equal to the weight in grams of ethereal salts (calculated as ethyl acetate) in the volume of liquor taken for the determination."

METHODS OF TANNIN ANALYSIS.

Page 78.—Omit all of 2 and insert the following:

(*a*) Shake 10 grams of the hide powder with 250 cc of water for five minutes; filter through muslin or linen; squeeze out thoroughly by hand; collect the hide powder, and repeat the operation three times with the same quantity of water. Pass the last filtrate through paper until a perfectly clear liquid is obtained. Evaporate 100 cc of the final filtrate in a weighed dish; dry at 100° until the weight is constant. If the residue amount to more than 10 mg, the sample should be rejected. The hide powder must be kept in a dry place and tested once a month.

(*b*) Prepare a solution of pure gallotannic acid by dissolving 5 grams in 1 liter of water. Determine the total solids by evaporating 100 cc of this solution and drying to constant weight. Treat 200 cc of the solution with hide powder exactly as described in paragraph 6. The hide powder must absorb at least 95 per cent of the total solids present. The gallotannic acid must be completely soluble in water, alcohol, acetone, and acetic ether, and should contain not more than 1 per cent of substances not removed by digesting with excess of yellow mercuric oxid on the steam bath for two hours.

Omit all of 3 and insert the following:

Dry 3 grams of the sample, if it be an extract, in a flat-bottomed dish not less than two and one-half inches in diameter, to constant weight. Of barks and similar substances take two grams.

Omit all of 4 and insert the following:

In the case of barks, woods, leaves, etc., use such an amount of the material as to give about 1 gram of total solids per 100 cc of solution, and extract in a Soxhlet or similar apparatus. In the case of extracts, use 20 grams, dissolve in 900 cc of water at 80°, let stand for twelve hours and dilute to 1,000 cc.

Under 5 first line after "and" insert: "without filtering."

Omit all of 6 and insert the following:

6. Determination of Soluble Solids.

Filter about 125 cc of the solution through a double folded filter (S. and S. No. 590, 15 cm) returning the filtrate through the filter once. Evaporate 100 cc as before.

Page 78, omit all of 7 and insert the following:

7. Determination of Tanning Substances.

Prepare 20 grams of hide powder by shaking for five minutes with 250 cc of water and straining through linen. Repeat the operation three times. Finally, remove as much water as possible by squeezing in a press. Transfer the pressed hide powder to a covered dish, and weigh. Dry a portion approximately equal to one-fourth of the whole to constant weight at 100°. Add the remainder of the hide powder to 200 cc of the original unfiltered solution and shake for ten minutes. Squeeze through linen, collect the filtrate, add 5 grams of kaolin free from soluble salts, stir well, and filter through a folded filter (S. & S. No. 590, 15 cm), returning the first 25 cc. Evaporate 100 cc as before. The weight of the residue must be corrected for the dilution caused by the water contained in the hide powder. The shaking must be done in some form of mechanical shaker. The simple machine used by druggists, and known as the "milk shake" is recommended.

Page 78, at the end of VIII–1, insert:

"Extracts must be heated to 50°, well shaken, and allowed to cool slowly to room temperature."

METHODS OF TANNIN ANALYSIS.

Add on page 78, after 7:

"8. Testing the Nontannin Filtrate.

"(a) *For tannin.*—Test a small portion of the clear nontannin filtrate with a few drops of a 1 per cent solution of gelatin. A cloudiness indicates the presence of tannin, in which case the determination must be repeated, using 25 grams of hide powder instead of 20 grams.

"(b) *For soluble hide.*—To a small portion of the clear nontannin filtrate add a few drops of the original solution, previously filtered to remove reds. A cloudiness indicates the presence of soluble hide due to incomplete washing of the hide powder. In this case, repeat the determination with perfectly washed hide powder."

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